भारतीय मानक
Indian Standard

IS 17449 : 2022

मोनोमेरिक एमडीआई (एमएमडीआई) और पॉलिमरिक एमडीआई (पीएमडीआई) — विशिष्टि

(पहला पुनरीक्षण)

Monomeric MDI (MMDI) and Polymeric MDI (PMDI) — Specification

(First Revision)

ICS 71.080.99

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Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, coal and Related Products Division Council.

Methylene diphenyl diisocyanate (MDI) is a broad family of chemical substances which all share certain common structural features as a consequence of their common starting point from the initial manufacture of crude MDI. The entire MDI family includes all methylene diphenyl diisocyanate (MDI) substances with key structural similarities. This structural similarity is based on a common functional group, that is, the terminal moiety consisting of two aromatic rings joined by a methylene bridge and where the terminal aromatic ring has a single isocyanate group in either the para or ortho position to the methylene bridge. Any of the substances in this group will have at least one (and more usually two or more) of the previously described functional group. This standard applies only to the generic grade of Polymeric MDI (PMDI) used typically in rigid foam applications and separately to Monomeric MDI (MMDI). For the avoidance of doubt, this standard does not include other grades of Polymeric MDI like high functional MDI, MDI prepolymers or other modified MDI products or isocyanates which are derivatives of MDI.

Based on the recommendations of Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizer, the Committee has decided to formulate Indian Standard on pure MDI, polymeric or crude MDI.

The standard was first published in 2020. In this first revision, the title has been modified from ‘Pure MDI and crude or polymeric MDI — Specification’ to ‘Monomeric MDI (MMDI) and polymeric MDI (PMDI) — Specification’ as pure MDI is a generic term while monomeric MDI is technically right. Moreover crude MDI and polymeric MDI are not same, their specifications are different. Accordingly, based on the change in title, the changes are made in the standard. The characteristics of appearance and colour have been deleted from Table 1 and only one form is mentioned with modified value of isocyanate (NCO) content in Table 1. Also, the requirement of isocyanate (NCO) content and acidity have been modified in Table 2.

The following alternate test methods also exists for testing the requirements specified in Table 1 and Table 2. In case of dispute the methods mentioned in Table 1 and Table 2 shall be the referee methods.

Viscosity at 25 ºC	ASTM D 4889
Isocyanate (NCO) Content	ASTM D 5155
Acidity for Monomeric MDI (MMDI)	ASTM D 5629
Acidity for Polymeric MDI (PMDI)	ASTM D 6099

Safety instructions to be followed during testing are given in informative Annex E.

The composition of the Committee responsible for the formulation of this standard is given at Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MONOMERIC MDI (MMDI) AND POLYMERIC MDI (PMDI) — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements, the methods of sampling and testing for monomeric MDI (MMDI), including pure MDI and polymeric MDI (PMDI).

1.2 This specification covers only one grade of PMDI which is majorly used in rigid polyurethane application. This specification does not cover prepolymers, high functional, modified and other variants of MDI.

2 REFERENCE

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
1448 (Part 25/Sec 1) : 2018/ISO 3104 : 1994	Methods of test for petroleum and its products: Part 25 Transparent and opaque liquids, Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)
8768 : 2000	Method of measurement of colour in liquid chemical products platinum — Cobalt scale (*second revision*)
13360 (Part 11/Sec 11) : 1999/ISO 3219 : 1993	Plastics — Methods of testing: Part 11 Special properties, Section 11 Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate

3 REQUIREMENT

3.1 Description

The material shall be a white to light yellow solid or liquid in the case of monomeric MDI (MMDI) and brown to dark brown colour liquid in the case of polymeric MDI (PMDI).

3.2 The material shall also comply with the requirements given in Table 1 for monomeric MDI and Table 2 for polymeric MDI. In case monomeric MDI is in liquid form, colour shall be 200 Pt-Co units when tested according to IS 8768.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable drums or bulk tankers conforming to local transportation guidelines.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear and indelibly the following information:

a) Name of the material;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Month and year of manufacture;
d) Lot or batch number;

- e) Net weight; and
- f) Any other statutory requirements.

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Tests for the determination of all other characteristics given in Table 1 and 2 shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Composite Sample*

For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1 and Table 2.

6 TEST METHODS

6.1 Test shall be conducted as prescribed in col 4 of Table 1 and Table 2.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities, which affect the result of analysis.

Table 1 Requirements for Monomeric MDI

(*Clauses* 3.2, 5.2 , 5.3.1 *and* 6.1)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to Annex (4)
i)	Isocyanate (NCO) content, percent by Mass, *Min*	33.0	B
ii)	Acidity, percent by mass, *Max*	0.005	C

Table 2 Requirements for Polymeric MDI (PMDI)

(*Clauses* 3.2, 5.2,5.3.1 *and* 6.1)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to Annex (4)
i)	Appearance	Brown to dark brown colourliquid	A
ii)	Isocyanate (NCO) content, percent by Mass, *Min*	30.0	B
iii)	Acidity, percent by mass, *Max*	0.06	C
iv)	Viscosity at 25 ºC, mPa·s	150-250	D/IS 13360 (Part 11/Sec 11)/IS 1448 (Part 25/Sec 1)

ANNEX A

[*Table* 2, *Sl No.* (i)]

DETERMINATION OF APPEARANCE (VISUAL INSPECTION PROCEDURE)

A-1 GENERAL

This is a method for the determination of appearance of polymethylene phenyl isocyanate by visual inspection.

A-2 PRINCIPLE

The sample is examined visually to check the colour.

A-3 SAMPLING

The sample must be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to moisture before visual inspection as moisture reacts with polymethylene phenyl isocyanate to form solids.

A-4 PROCEDURE

Visually examine the sample to see if any haze or foreign particles are present. Also, check the colour of sample. It may be necessary to swirl the sample to stir up any material that has settled to the bottom of the sample bottle.

A-5 REPORT

Report the appearance of the sample colour.

ANNEX B

[*Table* 1, *Sl No.* (i) *and Table* 2, *Sl No.* (ii)]

DETERMINATION OF ISOCYANATE (NCO) CONTENT BY DI-*N*-BUTYLAMINE TITRATION

B-1 GENERAL

This method is applicable to the determination of isocyanate content in the range of 5 percent and above.

B-2 PRINCIPLE

The isocyanates are reacted with di-*n*-butylamine and the excess di-*n*-butylamine is back-titrated with hydrochloric acid. The isocyanate content is calculated from the di-*n*-butylamine used in the titration.

B-3 SAMPLING

The sample must be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with polymethylene phenyl isocyanate to form solids.

B-4 APPARATUS

B-4.1 Lunge Pipette or Any Weighing Device, suitable for weighing a liquid sample in the absence of air by difference to the nearest 0.001 g.

B-4.2 Balance, **Analytical**, having minimum detection of 0.1 mg.

B-4.3 Glass Burette, class A, 50 ml.

B-5 REAGENTS

Unless otherwise specified, AR grade chemicals shall be used.

B-5.1 di-*n*-butylamine, 2 N in dry toluene.

Take 328 ml di-*n*-butylamine in dry 1.0 l volumetric flask and dilute it up to the mark with toluene. Stir thoroughly for 15 min.

B-5.2 Isopropyl Alcohol

B-5.3 Hydrochloric Acid, 1.0 N standard.

Take 104 g concentrate hydrochloric acid in dry 1.0 l volumetric flask and dilute it up to the mark with distilled water.

B-5.4 Bromocresol Green Indicator, 0.1 g/100 ml methanol.

Dissolve 0.1 g bromocresol green indicator in 1.5 ml of 0.1 M sodium hydroxide (NaOH). Dilute it to 100 ml with distilled water.

B-5.5 Toluene

B-6 PROCEDURE

B-6.1 To a dry 500 ml Erlenmeyer flask, add 40 ml dry toluene. Transfer 50 ml of 2 N di-*n*-butylamine to the flask using a clean and dry 50 ml pipette. Immediately stopper the flask and cool it in an ice water bath to a temperature of 0-10°C. While the di-*n*-butylamine solution is cooling, transfer a portion of the sample from the sample container to sample vial which is equipped with a stopper. Weigh the vial containing the sample to the nearest 0.000 1 g. Using the medicine dropper, transfer 5.0 to 6.0 g of sample to the erlenmeyer flask. Keep the erlenmeyer flask in the ice-water bath during the entire sample addition. Slowly swirl the di-*n*-butylamine solution while adding the sample to prevent local heating, and hence, volatilization (loss) of the di-*n*-butylamine. To prevent loss of the di-*n*-butylamine, keep the flask stoppered at all times except when actually adding materials to the flask. Reweigh the sample vial to the nearest 0.000 1 g. Determine the sample mass by difference.

B-6.2 Loosen the flask stopper slightly and remove the flask from the ice-water bath. Allow the flask to warm up to room temperature, taking care that the stopper of the flask is loose so pressure will not be developed. Remove the stopper from the flask and add (with a measuring cylinder) 225 ml of isopropyl alcohol. Add approximately 1.0 ml of bromocresol green indicator to the flask. Using a clean 50 ml burette, titrate the excess di-*n*-butylamine with standard 1 N hydrochloric acid to end point (blue to a yellow colour which persists for at least 15 s).

B-6.3 Note and record the volume in ml of 1 N hydrochloric acid required for the titration. Read the burette to the nearest 0.05 ml. Be sure to use the meniscus reader to read the burette to the nearest 0.05 ml, after allowing proper time for burette drainage. Run 2 blanks at the same time in exactly the same manner but omitting the sample. Pipette the di-*n*-butylamine for the blanks and the samples, at the same time.

B-7 CALCULATION

Isocyanate content (NCO), percent =

$$\frac{(A-B)\times N\times 42.02\times 100}{1\,000\times W}$$

where

A = corrected volume hydrochloric acid for the blank, ml;

B = corrected volume hydrochloric acid for the sample, ml;

W = mass of sample, g; and

N = exact normality of the hydrochloric acid.

ANNEX C

[*Table* 1, *Sl No.* (ii) *and Table* 2, *Sl No.* (iii)]

DETERMINATION OF ACIDITY

C-1 GENERAL

This method is applicable to the analysis of organic isocyanates.

C-2 PRINCIPLE

The isocyanates are reacted with excess *n*-propanol to form urethanes. The acidic components release are absorbed in the reaction mixture and titrate potentiometrically with methanolic potassium hydroxide.

C-3 SAMPLING

The sample must be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with diphenyl methane diisocyanate to form solids.

C-4 APPARATUS

C-4.1 Automatic Titrator, complete with reference and glass electrodes.

C-4.2 Buffer Solutions, *p*H 4 and 7.

C-4.3 Beaker, 250 ml.

NOTE — Automatic instrument is also available in which all calculations and the printout of results is done automatically.

C-5 REAGENTS

Unless otherwise specified, AR grade chemicals shall be used.

C-5.1 Potassium Hydroxide, 0.01 N methanolic, standardized to ± 0.000 1 N.

Take 0.66 g potassium hydroxide (assay 85 percent) in dry 1.0 l volumetric flask and dilute it up to the mark with methanol. Stir thoroughly for 15 min.

C-5.2 Hydrochloric Acid, 0.01 N, standardized.

Take 1.042 g hydrochloric acid (assay 35 percent) in dry 1.0 l volumetric flask and dilute it up to the mark with methanol. Stir thoroughly for 15 min.

C-5.3 *n*-Propanol

C-6 PROCEDURE

Add 100 ml of *n*-propanol to 250 ml glass beaker. Prepare the titrator according to its operation manual.

If necessary, standardize instrument to *p*H 4 with buffer solution, while the solution is gently agitated. Immerse electrodes in *n*-propanol. Adjust solution to a *p*H of 5.0 with either 0.01 N alcoholic potassium hydroxide or 0.01 N hydrochloric acid very carefully. Remove electrodes from the solution. Add 2 to 5 g of the sample into beaker while agitating using suitable sampling device. Cover with a watch glass and continue agitation for 10 min. Immerse electrodes in sample solution and titrate with 0.01 N alcoholic potassium hydroxide, to the end point. The equivalence point will occur in the range of about 5.50 to 7.00 *p*H units.

C-7 CALCULATION

$$\text{Acidity, as percent HCl} = \frac{A \times N \times 3.647}{S}$$

where

A = volume of potassium hydroxide solution required, ml;

N = normality of potassium hydroxide solution; and

S = mass of sample, g.

ANNEX D

[*Table* 2, *Sl No.* (iv)]

DETERMINATION OF VISCOSITY OF POLYMETHYLENE PHENYL ISOCYANATE

D-1 GENERAL

The method is applicable to the measurement of the viscosity of polymethylene phenyl isocyanate.

D-2 PRINCIPLE

The drag on a spindle rotating at a constant speed while immersed in the material under test is measured by a calibrated spring.

D-3 SAMPLING

The sample must be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with diphenyl methane diisocyanate to form solids.

D-4 APPARATUS

D-4.1 Viscometer, of any suitable make or model.

D-4.2 Spindles, set of four, to cover viscosity range of 0 to 100 000 centipoises.

D-4.3 Viscosity Standard, of desired range (silicon oil).

D-5 PROCEDURE

D-5.1 Put the instrument on the table. Set the level of instrument using level indicator, which is mounted on the top of instrument. Insert the plug in main switch and switch 'on' the instrument wait for 5 min to warm up the instrument. The spindles are attached to the viscometer by screwing them to the lower shaft. Note that the spindles have a left-hand thread. The lower shaft should be held in one hand and the spindle screwed to the left. The face of the spindle nut and the matching surface on the lower shaft should be smooth and clean to prevent eccentric rotation of the spindle. Spindles can be identified by the number on the side of the spindle coupling nut. The viscometer may have a spindle entry code number to calculate viscosity, shear rate and shear stress values. Be sure that the contacting surfaces of the spindle couplings are clean before screwing together. This will insure perfect alignment of the upper and lower shafts.

D-5.2 Before readings are taken, the viscometer must be set to zero. This action is performed each time the power switch is turned on. The display window in the viscometers may guide through the procedure. Press 'select spindle' key and set the spindle code as per instruction manual. Press 'select display key' and set the unit of viscosity 'cPs or mPa.s' on display screen. Press 'Motor ON/OFF' key for motor start and stop for measurement of viscosity of sample. Now instrument is ready for determination of viscosity of the sample.

D-5.3 Take sample in beaker and dip the spindle in the sample up to mark on the spindle. After reaching the desired temperature of sample, press the motor On/Off key for measurement of viscosity. Set RPM in a way that torque is maximum. Viscosity of the sample is directly shown on the display screen in 'cPs or mPa.s' whichever is selected.

ANNEX E

(*Informative*)

E-1 SAFETY INSTRUCTIONS TO BE TAKEN DURING TESTING

E-1.1 General Instructions

a) Isocyanates, may cause local skin irritation and care must be taken to avoid contact. If a spill is encountered, wash the affected area promptly with soap and water.

b) The maximum allowable concentration for poly methylene phenyl isocyanate in air has been tentatively set at 0.1 ppm. Use adequate ventilation to keep the vapour concentration at a minimum.

E-1.2 Instructions for Determination isocyanate (NCO) Content in Polymethylene Phenyl isocyanate By di-*n*-butylamine Titration

Toluene is a flammable liquid. Keep sparks and flames away.

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
Chemical Engineering and Process Development Division, NCL, Pune	Dr C. V. Rode (***Chairman***)
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All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
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Member Secretary

MS ADITI CHOUDHARY
SCIENTIST 'B' (PCD), BIS

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